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Micro-texturing of DLC thin film coatings and its tribological performance under dry sliding friction for microforming operation

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Abstract

As an approach to enhance the tribological performance of diamond like carbon (DLC) coating films for dry microforming operations, the present study applied the micro-textured structures to the DLC films. Micro-texturing was realized by using stainless steel wire mesh as a masking during the ionized physical vapor deposition (I-PVD) of DLC films. Tribological performance of fabricated micro-textured structures with a width of 40 and 80µm and an interval of 30 and 40 µm were evaluated by the ball-on-disk type friction tester. To simulate the severe contact state of dry microforming operation, normal pressure of 1.2GPa was applied for 100,000 times rotations under unlubricated condition. As results, the micro-textured DLC films with structure size of 40µm showed the excellent stable variation with relatively low coefficient of friction (COF) of μ = 0.01-0.06, while the conventional non-textured DLC films showed the significantly large deviation with high COF of $\mu > 0.25$. In comparison between the different micro-texture sizes, the smaller structure size of 40 µm showed the lower COF than that of 80µm, although the larger amount of wear debris from DLC films were generated. Additional wear track observation after the friction tests revealed that the stable tribological properties of textured DLC films were attributed to the promotion of wear debris ejection, which can prevent the plowing inside the apparent area of contact.

Keywords: Microforming; Dry friction; Diamond like carbon; Micro-texturing

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1. Introduction

Lubricant-free microforming process is a strong demand from the industry. Due to the low effect of lubricant in microscale dimensions (Engel, 2006), the tribological issues in microforming confronts with further severe problems to enhance the performance of microforming dies. In particular, tool life of the micro-dies has not been well satisfied the industrial requirement of endurance with more than 100,000 cycles.

Against these backgrounds, a number of applications of hard film coatings on microforming die substrates have been gradually increasing. Particularly, diamond like carbon (DLC) films has been received a great attention for its excellent tribological performance, such as low friction coefficient and high wear resistance under dry friction (Grill, 1997). First application was reported by Yang et al. (2004), who applied to the micro-extrusion die using pulse plasma chemical vapor deposition (CVD) method. Similarly, the friction properties of DLC coated micro-dies were followed by Krishnan et al., (2007) for micro-extrusion process and by Fujimoto et al. (2006), Aizawa et al. (2010), Hu et al. (2011) and by Wang et al. (2013) for micro-sheet metal forming process.

In spite of these developments above, the problems in low adhesion strength of DLC films with substrate has been remained as a technical challenges to endure under the high impact surface pressure in forming process. As an approach to enhance the adhesion properties of DLC films, surface texturing by segmentation of DLC films, which is possible to decrease inner stress and applied strain in deposited films, was proposed by Aoki et al. (2004). They demonstrated the twice longer endurance for micro-textured DLC films compared with the non-textured DLC films.

In addition to this anti-delamination effect, surface texturing has further advantages in tribological properties. One is the well-known function of the local increase of lubricant supply by fluid reservoirs creation and also the increase of load carrying capacity by a hydrodynamic effect (Blatter et al., 1999). Another is the function of wear debris entrapment effect in the trench or valley of the textured geometries (Suh et al., 1994). Although a number of studies for the metal forming operation have been focused on the former effect under the hydrodynamic or boundary lubricated conditions, the interfacial effect of surface texturing under dry sliding friction has not been clearly demonstrated in view of the application to microforming dies. To design the dimensions and geometries of the texturing patterns for dry microforming operation, the effect on the wear debris generation and its entrapment behavior, which might be a crucial impact on the dry sliding properties, has to be discussed more in detail.

The present study focuses on the tribological performance of micro-textured DLC films under dry sliding friction. In view of the application to the microforming dies, tribological properties were investigated under the contact state as operated in microforming process. Firstly, the micro-textured DLC films were fabricated by masking with metallic mesh during the ionized physical vapor deposition (I-PVD). Then, the tribological properties of this micro-textured DLC films were evaluated by ball-on-disk type friction tests. Based on this evaluation, the effect of the texture size on the dry friction and wear performance were discussed in detail.

2. Experimental

2.1. Ionized physical vapor deposition of DLC films and its micro-texturing

Depositions were performed in an I-PVD system (NPS-330S from Nanotec Corp.) with a usable coating volume of ϕ150 mm × 150 mm. In this system, the direct current ion source, composed with a tantalum (Ta) filament and anode electrodes, was installed to ionize the C_6H_6 benzene gas for the DLC deposition.

As a material generally used for microforming dies, sintered tungsten carbide-cobalt (WC-Co) hard alloy, JIS: V20, with a size of 14 mm × 14 mm and a thickness of 5mm were used as substrates. Prior to the deposition, this WC-Co substrates were mirror polished and ultrasonically cleaned in acetone. Starting with a base pressure of $<5\times10^{-3}$ Pa, argon (Ar) gas of 10 sccm was introduced to initiate the Ar plasma to remove the contaminated layer on the substrates. The pretreatment was performed under a dc voltage of -1.5 kV for 30 min. Subsequently, the DLC deposition was carried out in a C_6H_6 benzene atmosphere at 200 °C. The C_6H_6 flow was set to 1.5 sccm to maintain a total pressure of 2.6×10^{-1} Pa. Throughout the deposition, the substrate bias voltage was maintained at −1 kV. The conditions of whole process above were summarized in Table 1. Besides, a Raman spectrum of the obtained DLC film is shown in Fig. 1. The typical DLC spectrum consisted with a broad peak at approximately

1550cm−1, which is fitted with 2peaks of G- and D-band, was successfully obtained (Tamor, 1994). Additional measurements of nanoindentation tests revealed that the Young's modulus and the nanoindentation hardness of obtained DLC films were 287.3 ± 9.5 GPa and 27.8 ± 1.1 GPa, respectively.

To fabricate the micro-textured structure, according to the report of Aoki et al., metallic mesh was placed on the substrate as a masking plate during the DLC deposition. Stainless steel wire mesh with the wire diameter of 30 μm was used. To compare the effect of structure size of micro-texture, wire mesh with different grid interval of 80 and 40 μm was chosen. Fig. 2 shows the appearance of the stainless steel wire mesh used in the present study. Deposition conditions were the same as those in the case of the non-textured DLC films as shown in Table 1. The geometrical dimensions of the textured structure were measured by an atomic force microscopy (AFM).

Table 1. Conditions of DLC deposition by I-PVD process.

Pre-treatment of substrate		Ar^{+} bombard dc -1.5kV for 30min
Gas species		C_6H_6
Gas Flow Rate	(sccm)	1.5
Gas Pressure	(Pa)	2.6×10^{-1}
Bias Voltage	(kV)	-1.0
Filament Current	(A)	30
Anode Voltage	(V)	30
Temperature	(˚C)	200
Deposition time	(h)	4.5

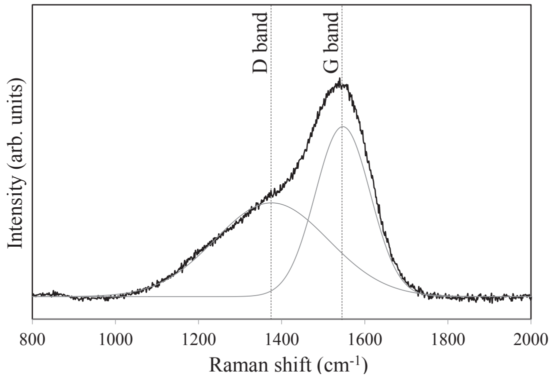
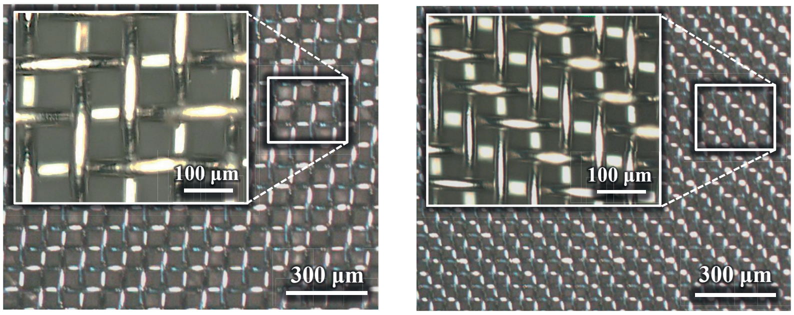


Fig. 1. Raman spectrum of obtained DLC films on a WC-Co substrate.



(a) Mesh with grid-interval of 80μm (b) Mesh with grid-interval of 40μm

Fig. 2. Appearance of stainless-steel wire meshes for masking during DLC deposition.

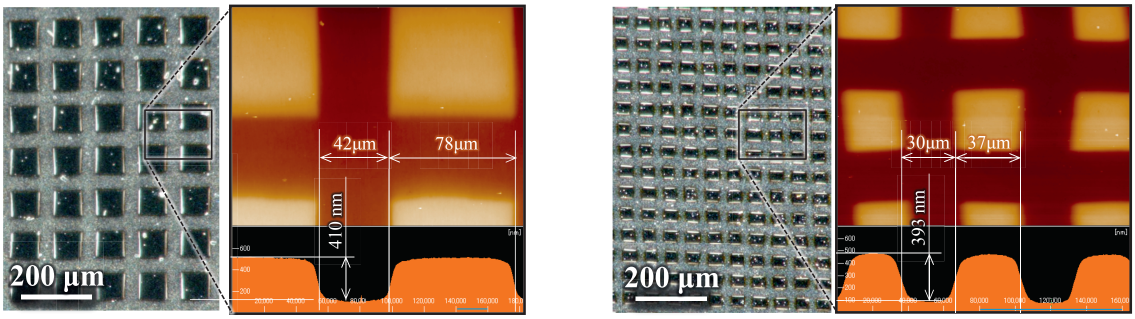
2.2. Tribological evaluations

To characterize the basic tribological properties of fabricated non-textured and micro-textured DLC films under dry sliding conditions, a ball-on-disk type tribology tests were carried out with a commercial tribometer (TRIBOMTER, CSM Instruments). A constant normal force of 5N, which corresponds to an initial Hertzian mean contact pressure of about 1.2 GPa and elastic contact diameter with 114 μm, was applied for the all testing samples. This contact pressure is equivalent to the one generally mentioned in the severe contact state in metal forming operation, such as ironing or forging process. As a counterpart of contacting pair, a 6 mm JIS: SUJ2 steel ball was used. The tests were carried out under unlubricated condition and with a linear speed of 100 mm/s at room temperature in an ambient air condition for 100,000 laps, which corresponds to the industrially required number as a tool life for forming dies. Wear tracks of the substrates and sliding balls were observed by optical microscopy and the scanning electron microscopy (SEM) after the friction test. Moreover, chemical composition of the generated and adhered wear debris was locally analyzed by energy dispersive X-ray spectroscopy (SEM-EDX).

3. Results and discussion

3.1. Fabrication of micro-textured DLC Films

Fig. 3 shows the surface images of micro-textured DLC films obtained by an optical microscopy and AFM. The different grid-like structure was successfully obtained both for the mesh grid-interval of 80 and 40 μm. As shown in Fig. 3, the obtained structure dimensions for the grid-interval of 80 μm are the structure width of 78 μm and the interval of 42 μm, while for the grid-interval of 40 μm shows the structure width of 37 μm and the interval of 30 μm. The structure height is approximately 400 nm for both grid-interval conditions. Although the mesh with same wire diameter of 30 μm was used, the structure interval had a 10 μm difference. It may be due to the difference in ion flux at the near surface depending on the grid-interval. However, the structure size are well corresponded to the grid-interval of metal mesh. Accordingly, here we describe the micro-textured DLC films deposited with the 80 μm grid-interval mesh as textured-80 μm and that with the 40 μm grid-interval mesh as textured-40 μm.



(a) DLC films deposited with the mesh of 80μm grid-interval (b) DLC films deposited with the mesh of 40μm grid-interval

Fig. 3. Surface images of micro-textured DLC films obtained by optical microscopy(right) and AFM(left).

3.2. Evolution of coefficient of friction

Fig. 4 shows the evolution of coefficient of friction (COF) during the 100,000 times rotations of ball-on-disk type friction tests for non-textured and micro-textured DLC films. The high COF with relatively large deviation is shown for the non-textured films. Particularly, after the 40,000 laps, the deviation of COF becomes larger and it exceeds μ = 0.25. While for the textured DLC films, the variation of COF is small and the stable evolution is observed both for the 80 and the 40μm textured films. Additionally, the relatively low COF value is indicated for textured condition, which shows the μ = 0.05 -0.08 for textured-80μm, and the μ = 0.01 -0.06 for textured-40 μm.

To investigate the cause of this difference in friction properties, the wear tracks of the DLC films and the counterparts of SUJ2 balls are observed, as shown in Fig. 5. The largest width of the wear track is shown for the non-textured DLC films, whereas the textured-80 μm films indicate the smallest. Corresponding to this result, the largest diameter of worn area of the SUJ2 ball is observed for non-textured condition. Furthermore, the accumulation of the worn material is observed at the backward side from the direction of sliding. Since the large amount of oxygen (O) and Iron (Fe) contents was detected by the local analysis by EDX, this accumulated material appears to be the aggregate of iron oxides. During the dry sliding friction, newly formed surface and wear debris from the SUJ2 balls rapidly reacts with oxygen atmosphere and it builds the iron oxide. And these hard and brittle particles drop from the surface and plow the contacting pair or itself, which is well known as oxidative wear (Quinn, 1980). The repetition of trap and drop-off of these oxides particles at the contact surface strongly affects the deviation and stability of the friction properties (Suh, 1981). In fact, as observed in Fig. 5 (d), the size of this oxides accumulation for non-textured surface shows extremely large diameter of 299μm, while that for the textured surface is almost the same size with the worn area at the center. Consequently, the high COF and relatively large deviation of the non-textured surface is strongly attributed to the trap and drop-off behavior of oxidative wear debris from the SUJ2 steel balls. In other words, textured structure appears to promote the ejection of these wear debris, so that it can suppress the plowing by these hard and brittle oxides particles.

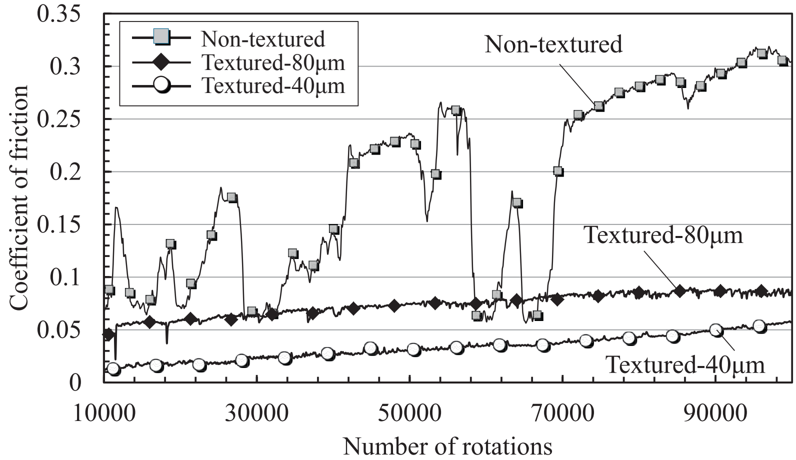


Fig. 4. Evolution of COF of non-textured and micro-textured DLC films under dry sliding friction during ball-on-disk friction test. [operating conditions: sliding speed:100 mm/s, initial Hertzian pressure: 1.2 GPa]

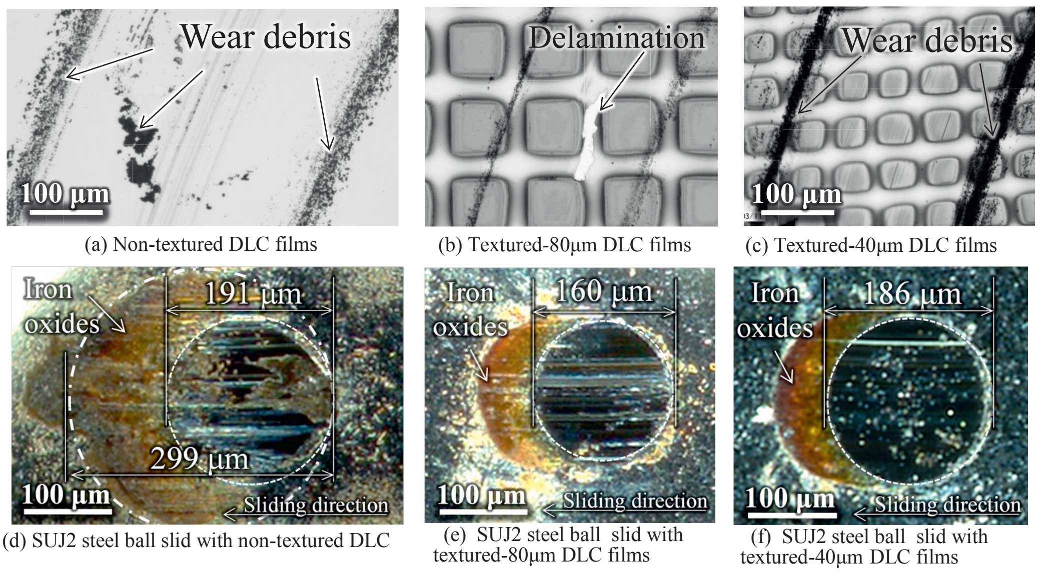


Fig. 5. Optical surface images of wear tracks of un-textured and micro-textured DLC films and its counterparts of SUJ2 balls after the ball-on-disk friction test with 100,000 laps.

Meanwhile, in comparison between the different texture sizes, the smaller texture size with the textured-40 μm sample shows the larger wear track width and the diameter of the worn area of the SUJ2 balls. Additionally, the amount of wear debris beside the edge of the wear tracks on films is also larger for the textured-40 μm sample (Fig. 5(c)). According to the EDX analysis, these accumulated wear particles are found to be as carbon (C) contents, which appears to be generated from the DLC films. Thus, it can be considered that this larger volume of wear in the textured-40 μm samples is attributed to the higher normal pressure due to the smaller texture structure. However, since the relative number of the single textured structure inside the apparent contact area for textured-40 μm sample is more than the larger texture of 80 μm, the probability of the ejection of wear debris from the contact interface seems to be higher. In fact, inside the calculated Hertzian contact diameter of 114 μm, only one single textured structure of 80 μm can be exists. Thus, the larger structure would easily trap the wear particles inside the area of contact, and the plowing by these particles might increase the frictional resistance. Actually, a partial delamination of the DLC films, which seems to be attributed to the plowing by the wear debris grown at the interface, is observed through the wear track of the textured-80 μm samples (Fig. 5(b)). Consequently, the smaller textured structure of 40 μm plays an important role to promote the ejection of wear debris and it contributes to decrease the coefficient of friction. In contrast, since the texture size of the 80 μm is almost the same as the apparent contact area, the probability of the ejection of wear particles becomes lower and it results in the higher coefficient of friction.

4. Conclusions

In this report, the micro-textured DLC films was fabricated by using metallic mesh during the I-PVD process. In view of the application to the microforming dies, its tribological characteristics under dry sliding friction were demonstrated in comparison with the conventional non-textured DLC films. Following conclusions may be drawn:

(1) Micro-textured structure with a width of 40 and 80 μm and an interval of 30 and 40 μm was successfully fabricated.
(2) The textured DLC films with structure size of 40 μm showed the excellent stable variation with relatively low COF of 0.01-0.06 during the 100,000 laps rotation under the dry sliding friction and the severe contact pressure of 1.2 GPa.
(3) Although the larger amount of wear volume was observed for the smaller texture size of 40μm condition, the lower COF than the larger texture size of 80μm was obtained due to its higher performance in wear debris ejection, which can prevent the plowing inside the area of contact.

Consequently, it was demonstrated that the micro-textured structure plays an important role as a space to prevent the wear debris from trapping in the area of contact and to promote its ejection. The quantitative evaluation to design the appropriate geometrical structures specified for dry microforming operation would be our future task.

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